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CALIBRATION STANDARDS AND. (U) JOINT OIL ANALYSIS  
PROGRAM PENSACOLA FL TECHNICAL SUPPORT CEN. B L JONES  
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MICROCOPY RESOLUTION TEST CHART  
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TITLE: DETERMINATION OF THE SHELF-LIFE OF OIL ANALYSIS CALIBRATION STANDARDS AND EVALUATION OF A LABORATORY SHAKER FOR USE WITH OIL CALIBRATION STANDARDS

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FORWARD

This report contains the results of a study conducted to determine the maximum usable shelf-life of oil calibration standards used within the Joint Oil Analysis Program (JOAP). In addition, the study included the extent and mode of shaking, manual or mechanical, required to produce a homogeneous oil calibration standard which has been in storage for extended periods. An increase in shelf-life will result in less wastage resulting from the requirement to dispose of over age standards that have exceeded current shelf-life limits while in supply depots or held by JOAP laboratories.

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DETERMINATION OF THE SHELF-LIFE OF OIL CALIBRATION STANDARDS  
AND  
THE EVALUATION OF A SHAKER FOR OIL CALIBRATION STANDARDS

I. INTRODUCTION:

The assigned primary goal of this study was to determine the maximum useable shelf-life of oil calibration standards required to standardize the oil analysis spectrometers used in the Joint Oil Analysis Program. A restudy of the shelf-life limits is deemed advisable due to the lapse of time since the assignment of the present shelf-life limit, evolutionary changes in the standards formulation, and the lack of current data to support the laboratory work upon which the current shelf-life assignments are based.

Secondary purposes were to determine the extent and mode of shaking required to produce a homogeneous oil calibration standard after storage for extended periods, and to determine whether manual or mechanical shaking is the preferred method for field laboratories.

In conjunction with the mechanical shaking requirement determination, the TSC was tasked to conduct a study of off-the-shelf laboratory type mechanical shaking equipment to determine if these smaller portable shakers were more suitable for use by field laboratories than the large standard stationary paint shakers available at some operating sites.

The oil calibration standards used in the Joint Oil Analysis Program (JOAP) are blended mixtures of metallo-organic concentrates in pure hydrocarbon oil (MIL-L-6082, Gr 1100 without additives). The standards are blended in concentrations of 3, 10, 30, 50, 100 and 300 parts per million (PPM) levels for JOAP laboratories. Standards with concentration levels of 500, 700 and 900 PPM are also blended for special application. The 3, 10, 30, and 50 PPM levels contain 0.48 percent by weight of a stabilizer and the 100 and 300 PPM levels contain 0.12 percent by weight of a stabilizer. The current oil calibration standards contain specified amounts of fourteen (14) elements: iron (Fe), silver (Ag), aluminum (Al), chromium (Cr), copper (Cu), magnesium (Mg), sodium (Na), nickel (Ni), lead (Pb), silicon (Si), tin (Sn), titanium (Ti), molybdenum (Mo), and zinc (Zn).

All oil calibration standards except the zero (0) PPM are assigned a shelf-life at manufacture and standards older than 18 months since manufacture must be laboratory tested and compared with a current standard of the same concentration prior to approval for continued use. Following testing, shelf-life may be extended for one six month period, for a maximum service shelf-life of 24 months since date of manufacture.

There is some evidence to support the conclusion that during storage the metallo-organic compounds settle out at an unknown rate. This necessitates that all oil calibration standards, except the 0 PPM, be shaken when received from supply and one day prior to use. At present there is no standard procedure or equipment specified for assuring a homogeneous mixture by shaking. Consequently, the oil calibration standards are currently either shaken manually prior to use or not shaken at all.

Numerous activities have reported receipt of over age oil

calibration standards from the local supply activity. Other reports indicate that some JOAP laboratories continue to use oil calibration standards that have exceeded the 18 month shelf-life specified in the JOAP manual (NAVAIR 17-15-50, TM 38-301-1, T.O. 33-1-37). The use of over age or inadequately shaken oil calibration standards for spectrometer standardization may have adverse effects upon standardization of the instrument and consequently up on the accuracy of operational oil analysis results.

## II. OBJECTIVES:

### A. Objective One: Shelf-life Determination.

1. Determine viable shelf-life of standards by laboratory testing of standards that have exceeded the assigned shelf-life limits. To be judged acceptable, outdated standards must provide spectrometer readings within the quality assurance (QA) tolerances as compared to current standards as specified in the JOAP Manual.

2. Determine the effect of high temperature storage on the shelf-life of standards since no data is currently available. It was determined that it was not necessary to perform cold storage testing because the supplier of the metallo-organic concentrates, as well as two major research laboratories have stated that a cold storage environment has little or no affect on the shelf-life stability of oil calibration standards.

### B. Objective Two: Shaking Requirements.

1. Determine requirements for shaking by comparing the ability of hand shaking as opposed to mechanical shaking to ensure homogeneous standards. Homogeneity is to be measured by comparing the spectrometer readings of standards of various ages, both within and beyond the shelf-life limits, that were either shaken or not shaken, to the readings of current reference standards. Determine at what age (time since manufacture) that shaking becomes necessary, that is, when settling first begins to affect spectrometer results.

2. If mechanical shaking proves to be beneficial, determine extent of shaking (speed, oscillations, etc.) and time of shaking required. Evaluate various off-the-shelf laboratory shaking equipment available and prepare a proposed procurement specification for laboratory equipment that will best meet the requirement.

## III. TEST METHODOLOGY:

A. Spectrometric Analysis: Ten analyses of each concentration, alternating with ten analyses of the primary reference standard, were performed for each oil calibration standard.

B. Statistical Calculations: Each element was compared at the 95% confidence level in accordance with the following:

$$T = \frac{\bar{x}_1 - \bar{x}_2}{\sqrt{\frac{S_1^2 + S_2^2}{N}}}$$

$$F = \frac{S_1^2}{S_2^2} \text{ or } \frac{S_2^2}{S_1^2} \quad (\text{where the larger number is the numerator})$$

$$LB = (\bar{x}_1 - \bar{x}_2) - t_{95} \sqrt{\frac{S_1^2 + S_2^2}{N}}$$

$$UB = (\bar{x}_1 - \bar{x}_2) + t_{95} \sqrt{\frac{s_1^2 + s_2^2}{N}}$$

AI =  $(Y - \bar{X})$  or  $(\bar{X} - Y)$  (the value for AI is always positive)

$$RI = \sqrt{\frac{N (\sum x_{\bar{X}}^2) - (\sum x_1)^2}{N (N-1)}}$$

AI = Accuracy Index - A test performed by finding the difference between the expected concentration of the sample and the mean value of the 10 replications of the analyses and comparing with a historically developed maximum allowable accuracy index.

RI = Repeatability Index - A test performed by comparing the standard deviation of the results from the 10 replications of the analyses with a historically developed maximum allowable repeatability index.

F = F Test - A standard statistical test comparing the ratio of the sample standard deviations with the 95th percentile of the F distribution.

T = T Test - Standard statistical test comparing the T statistic with the 95th percentile of the T distribution.

PT = Practical Tolerance - A test for practical significance based on expanding the T-test 95% confidence limits by a historically developed amount necessary to achieve consistency with chemical accuracy required to make valid maintenance recommendations.

LB = Lower boundry for practical tolerance test.

UB = Upper boundry for practical tolerance test.

Y = Nominal value of the standard in PPM.

$\Sigma$  = Summation of the analytical data

$x_i$  = Individual analysis results

N = Number of consecutive analyses

X = Average value of N consecutive analysis

$s_i$  = Standard deviation of reference sample

$s_2$  = Standard deviation of test sample

$\bar{x}_1$  = Mean of reference sample

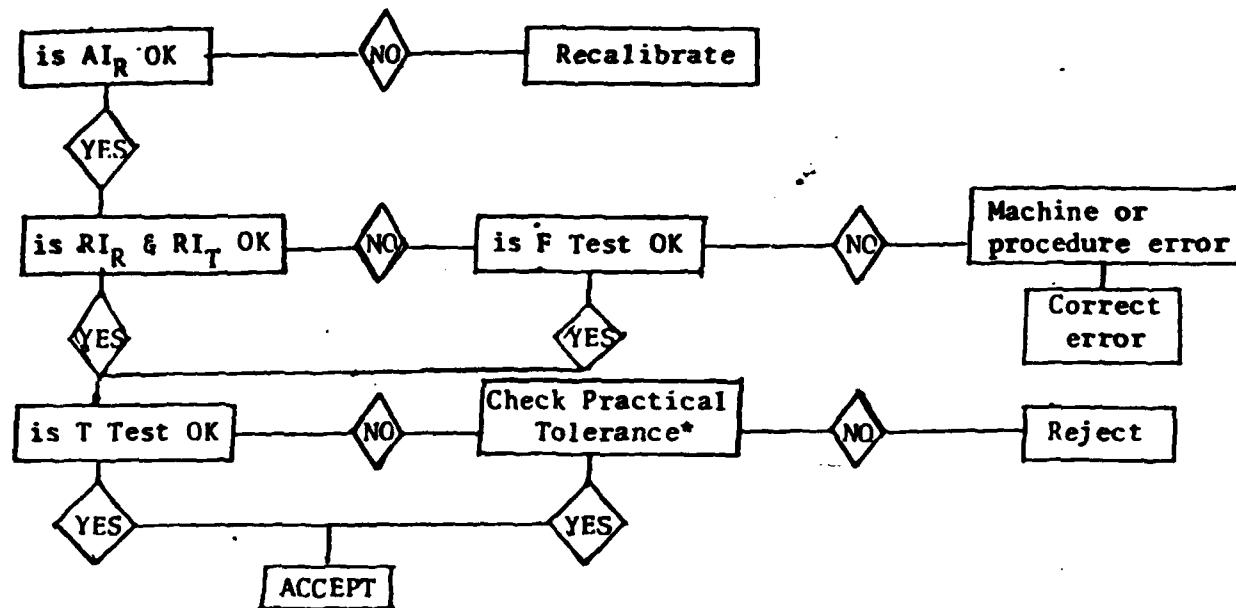
$\bar{x}_2$  = Mean of test sample

$t_{95}$  = 95% confidence level

#### C. Data Evaluation:

1. The spectrometric data for each concentration of each element were analyzed by application of the statistical tests listed above and the following flow chart.  $AI_R$  is accuracy index of reference standard,  $RI_R$  is repeatability index of reference standard, and  $RI_T$  is repeatability index of test samples:

SEE ATTACHMENT



\*Practical Tolerance: Obtained by taking 1/2 the mean difference (MD) and adding to the lower boundary or the upper boundary. If the value passes through "0", the standard is acceptable.

2. The following table is an example of the test rejections by concentrations for each element. The table is annotated to show whether rejections were due to high or low readings:

TABLE 1

PPM	Fe	Ag	Al	Cr	Cu	Mg	Na	Ni	Pb	Si	Sn	Ti	Mo	Zn
3							Rh							
10					R1									Rh
30	R1													Rh
50						Rh								
100								R1						
300			Rh											

Rh = Reject high

R1 = Reject low

D. Equipment/Supplies:

1. Equipment

- a. Baird Spectrometer, A/E35U-3
- b. Mechanical Shakers
  - (1) Burrell, Model 75, Wrist Action
  - (2) Red Devil Paint Conditioner, Model No. 5033X
- c. Oven, Blue M, Model OV-510A-Z
- d. Mixer, Triumph Model L-3-60

2. Supplies:

- a. Polyethylene bottles, 2 oz.
- b. Oil calibration standards within and beyond shelf-life limits (3,10,30,50,100,300 PPM)
- c. Recently prepared primary reference oil standards (3,10,30,50,100,300 PPM)
- d. Disc and rod electrodes, MIL-E-8971A
- e. Caps, sample, NSN 6640-01-042-6853

IV. PROCEDURE:

This study, with both objectives conducted concurrently, compared recently prepared primary reference oil standards with oil calibration standards of various ages, both within and beyond the current shelf-life limits. A laboratory mechanical shaker designed to accommodate the 8 oz. oil calibration standards bottle and a 2 oz. polyethylene bottle was used to shake the oils one day prior to analysis.

Samples of oil calibration standards of various ages were obtained from both supply sources and field activities. The primary reference oil standards were obtained from the JOAP-TSC inventory.

The dates since manufacture for the oil calibration standards varied from six (6) to sixty-four (64) months. Each concentration level of the over age oil calibration standard was not shaken, or was shaken with a laboratory mechanical shaker for a specified time and speed before analysis. Twenty replicate analyses, alternately comparing a recently prepared primary reference oil standard with the over age oil calibration standard for each concentration level, were made using the A/E35U-3 spectrometer. To be considered useable or serviceable the results had to agree with the QA criteria established for new primary reference oil standards as calculated in paragraph II.A.1.

The dates since manufacture for the oil calibration standards for the hot environmental tests varied from one (1) to twelve (12) months. Twenty replicate analyses, alternately comparing a recently

prepared primary reference oil standard with the oil calibration standard from each test interval, were made using the A/E35U-3 spectrometer. To be considered useable or serviceable, the results from the oil calibration standards had to agree with the quality assurance (QA) criteria established for new primary reference oil standards as calculated in paragraph II.A.1.

**V. RESULTS:**

**A. Unshaken.** Comparison tests were made on each concentration level (3, 10, 30, 50, 100 and 300 PPM) of oil calibration standard without shaking with recently prepared primary reference oil standards that had been shaken and allowed to settle overnight or until all foam and air bubbles had dissipated. The results are shown in Table 2.

One would expect the heavier more dense elements to settle more readily than the lighter less dense elements. This apparently occurs since more rejections are attributed to lower values (R1).

Nine elements (Fe, Al, Cr, Cu, Mg, Na, Ni, Si and Sn) are stable up to 36 months in all concentrations without shaking. Lead (Pb) is stable up to 33 months in all concentrations without shaking. Three elements (Ag, Ti, Zn) are stable up to 30 months in all concentrations without shaking. Molybdenum (Mo) is stable up to 22 months in 3 PPM concentrations without shaking and stable up to 30 months in all other concentrations without shaking.

Lower Ag and Ti values were expected in oil calibration standards packaged prior to January 1983 because of unstable concentrates used. That is, Ag was approximately 5% low and Ti was approximately 14% low.

**B. Shaken.** Comparison tests were made on concentration levels (3, 10, 30, 50, 100 and 300 PPM) of oil calibration standards which had been shaken at maximum speed with a mechanical shaker for periods of 10, 30, 60 and 120 minutes. After shaking and allowing to settle overnight or until all foam and air bubbles had dissipated, they were compared with recently prepared primary reference standards that had been shaken and allowed to settle as stated above. The results are shown in Table 3.

Five elements (Fe, Al, Cr, Cu and Ni) are stable for up to sixty-four months for all concentrations after adequate shaking. Ten elements (Fe, Al, Cr, Cu, Mg, Na, Ni, Si, Sn and Zn) are stable for up to 36 months for all concentrations after adequate shaking. Silver (Ag) is stable for up to 33 months for the 3 PPM oil concentration after adequate shaking and stable up to 36 months for all other concentrations after adequate shaking. Lead (Pb) is stable up to 33 months for all concentrations after adequate shaking. Titanium (Ti) is stable up to 32 months for all concentrations after adequate shaking. Molybdenum (Mo) is stable up to 30 months for all concentrations after adequate shaking.

Adequate shaking is defined as being shaken at the maximum control for a maximum of 60 minutes with a mechanical shaker equivalent to the Burrell Wrist Action Model 75 shaker.

The rejects for the higher values (Rh) for Ag, Pb, Sn and Mo were observed in older samples and for samples that had been shaken for 120 minutes (2 hours). The higher values for Na are attributed to the use of a base oil that was contaminated with up to 2 PPM Na.

A comparison of oil calibration standards are shown in Table 4. The rejects, high or low, cannot be attributed specifically to time since manufacture, but can probably be attributed to any one, or combinations of the following:

1. Instability of concentrates
2. Fluctuation of the A/E35U-3 spectrometer
3. Inadequate shaking

C. Effect of Hot Environment. We were not aware of any test method (ASTM, Fed Test Method, etc.) for determining the stability of metallo-organic oil calibration standards in hot environments. Consequently, the following method was used:

A bottle of each concentration level (3, 10, 30, 50, 100 and 300 PPM) of oil calibration standards (1 month to 12 months) was thoroughly shaken with a mechanical shaker for 30 minutes. After setting overnight, or until all foam and air bubbles had dissipated. Seven (7) 2 oz. bottles were filled with each concentration level. One bottle of each concentration level was compared with a recently prepared reference oil standard that had been shaken and allowed to settle as noted above. Six (6) 2 oz. bottles of each concentration level were placed in an oven heated to 130 F. Two (2) bottles of each concentration level were removed from the oven after 15, 30, and 45 days. One was shaken for 30 minutes with a mechanical shaker and allowed to settle as above, then compared with a recently prepared primary reference standard. The other bottle of each concentration level, when removed from the oven after 15, 30, and 45 days, but not shaken, was allowed to cool overnight and then compared with a recently prepared primary reference standard.

The results are shown in Table 5. The only failures from the effect of a hot environment was the 50 PPM (B25/15D/12M/S and B25/15D/12M/NS) concentration level after heating 15 days at 130 F and the 100 PPM (B63/30D/8M/S and B63/30D/8M/NS) concentration level after heating 30 days at 130 F. However, these same samples (B25/12M/S), B63-100/6M/S and B63/12M/120MB) are acceptable when shaken, but not when subjected to a hot environment (Table 3).

#### VI. CONCLUSIONS:

A. Oil calibration standards are stable up to 30 months without shaking for all concentration levels, except the 3 PPM. The 3 PPM concentration level is not stable for more than 22 months without shaking because of molybdenum.

B. Oil calibration standards are stable up to 30 months for all concentration levels with adequate shaking as defined in paragraph V.B. Here, mechanical shaking is required in order to assure uniform controlled and standardized conditions throughout the JOAP community since manual shaking cannot be controlled or standardized at field/operational laboratories.

C. The storage or subjection of oil calibration standards to a hot environment (130 F) appears to have only a slight effect.

#### VII. RECOMMENDATIONS:

A. Delete molybdenum (Mo) from oil calibration standards and extend the shelf-life to 30 months for all concentration levels with no shaking required.

B. Require each bottle of oil calibration standard with a manufacture date of twelve (12) months or more, and which contains molybdenum (Mo) be mechanically shaken one (1) hour the day prior to use.

Oil calibration standards should be administratively disposed of as used oil in accordance with local regulation when manufacture date of thirty (30) months is reached.

C. Change the label for the oil calibration standards to read:

1. Shelf-life 30 months
2. Delete retest data requirement

above

3. Mechanically shake at least 1 hour, one day prior to use
4. Do not store or subject to hot environments of 130° F or

TABLE 2. CALIBRATION STANDARDS UNSHAKEN

PPN	SAMPLE NO.	MONTHS	TIME SHAKEN	MINUTES	Fe	Ag	A1	Cr	Cu	Mg	Na	N1	Pb	Si	Sn	T1	Mo	Zn	Status
3	B40/NH/6M/NS	6		0														A	
3	B25-3/22M/NS	22		0					R1								R1*	R1	F
10	B34-10/8M/NS	8		0															A
10	IB73-10/64M/UP	64		0					R1								R1	R1	F
10	IB73-10/64M/LO	64		0						R1							R1	R1	F
10	MB11/33M/NS	33		0						Rh*							R1		F
30	B26/12M/NS	12		0															A
30	B25-30/48M/TOP	48		0													R1*		A*
30	B25-30/48M/BOT	48		0													R1*	R1	A*
30	B31/42M/NS	42		0													R1*		A*
50	B25/NH/12M/NS	12		0													R1*		A
50	B16-50/30M0/NS	30		0					R1								R1*	R1	F
50	B16/30M/36HNS	30		0					R1								R1*	R1*	F
100	B63/NH/8M/NS	8		0					Rh*								R1	R1*	A
100	B9-100/36M/NS	36		0					Rh								R1	R1	F
100	B16/37M/NS	37		0					R1								R1*	R1	F
100	B33/30M/NS	30		0													R1	R1	A

Rh - Reject, high  
 R1 - Reject, low  
 A - Accept  
 F - Fail

\*Borderline but acceptable

(1) Heated 36 hours at 130°F  
 (2) Zinc was not in this standard.

TABLE 2. (cont)

PPN	SAMPLE NO.	MONTHS	TIME SHAKEN MINUTES	TESTS												Status	
				Fe	Ag	Al	Cr	Cu	Mg	Na	Ni	Pb	Si	Sn	Ti	Mo	
100	BS9/7MO/NS	7	0														R1*
300	IB60/NH/1M/NS	1	0														A

Rh - Reject, high  
R1 - Reject, low  
A - Accept  
F - Fail

\*Borderline but acceptable

TABLE 3. CALIBRATION STANDARDS, SHAKEN

RH	- Reject,	high
RI	- Reject,	low
A	- Accept	
E	- Fail	

\*Borderline but acceptable

\*\*Only one bottle; shaken, reshaken after each analysis.

- (1) Heated 36 hours at 1300°F
- (2) Analyzed 1 day apart
- (3) Zinc not added to this sample

TABLE 3. (Cont)

PPN	SAMPLE NO.	MONTHS	TIME SHAKEN MINUTES	Fe	Ag	A1	Cr	Cu	Mg	Na	N1	Pb	Si	Sn	T1	Mo	Zn	Status
30	B17/22M/120/B	22	120, Burrell												Rh*		A*	
50	B25-50/12M/S (1)	12	30, Red Devil												R1*	R1*	A*	
50	B16/30M/36HS	30	30, Red Devil												R1*	Rh	R1*	F
50	B16/34M/120M/B	34	120, Burrell												Rh	R1	R1*	F
100	B63-100/6M/S	6	30, Red Devil												Rh	R1	R1*	F
100	B63/12M/120MB	12	120, Burrell												Rh	R1	R1*	A
100	B44/26M/120MB	(2)	120, Burrell												Rh	R1	R1*	A
100	B44/26M/120MB	26	120, Burrell												R1*	R1*	Rh	A*
100	B31/32M/60MB	32	60, Burrell												Rh	R1*	R1*	A*
100	B31/32M/120M/B	32	120, Burrell												R1*	R1	R1*	F
100	B27/34M/60MB	34	60, Burrell												R1*	R1*	R1*	A*
100	B27/34M/120M/B	34	120, Burrell												R1*	R1*	R1*	A*
100	B27/36M/120MB	36	120, Burrell												R1	R1	R1*	F
100	B16/37M/S	37	30, Red Devil												R1*	R1*	R1*	A*
100	B16/37M/60M/B	37	60, Burrell												R1*	R1*	R1*	A*
100	B16/37M/120M/B	37	120, Burrell												R1*	R1*	R1*	F
100	B9-100/38M/S	38	30, Red Devil												Rh	R1*	Rh*	F

Rh - Reject, high  
 R1 - Reject, low  
 A - Accept  
 F - Fail

\*Borderline but acceptable

TABLE 3. (cont)

P/N	SAMPLE NO.	MONTHS	TIME SHAKEN	MINUTES												Status	
				Fe	Ag	Al	Cr	Cu	Mg	Na	Ni	Pb	Si	Sn	Ti	Mo	
300	1B60/300/1M/S	1	30, Red Devil														A
300	B8/36M/120M/B	36	120, Burrell														A

Rh - Reject, high  
 R1 - Reject, low  
 A - Accept  
 F - Fail

\*Borderline but acceptable

TABLE 4. COMPARISON OF SHAKEN & UNSHAKEN OIL CALIBRATION STANDARDS

PPM	SAMPLE NO.	MONTHS	TIME SHAKEN MINUTES	Fe	Ag	Al	Cr	Cu	Mg	Na	Ni	Pb	Si	Sn	Ti	Mo	Zn	Status
3	B40/NH/6M/NS	6	0														A	
3	B40-3/6M/S	6	30, Red Devil														A	
3	B25-3/22M/NS	22	0	R1												R1*	F	
3	B25/30/22M/RD	22	30, Red Devil													R1*	A*	
3	B25/30/22M/B	22	30, Burrell													R1*	A	
10	MB11/33M/NS	33	0													R1	F	
10	B14-11/33M/10B	33	10, Burrell	Rh*												R1*	A*	
10	B11/36M/120M/B	36	120, Burrell	Rh	Rh*											Rh	F	
30	B26/12M/NS	12	0													Rh	Rh*	
30	B26-30/8M/S	8	30, Red Devil													Rh*	F	
30	B31/42M/NS	42	0													R1*	A	
30	B31/42M/10	42	10, Burrell													R1	A*	
30	B31/42M/30	42	30, Burrell													R1	F	
50	B25/NH/12M/NS	12	0													R1*	A	
50	B25-50/12M/S	12	30, Red Devil													R1*	A*	
100	B16/37M/MS	37	0	R1		R1*		R1*		R1		R1*		R1*		R1	F	
100	B16/37M/S	37	30, Red Devil													R1*	A*	

Rh - Reject, high  
R1 - Reject, low  
A - Accept  
F - Fail

\*Borderline but acceptable

TABLE 4. (cont.)

PPM	SAMPLE NO.	MONTHS	TIME SHAKEN MINUTES	Fe	Ag	Al	Cr	Cu	Mg	Na	Ni	Pb	Si	Sn	Ti	Mo	Zn	Status
100	B16/37M/60M/B	37	60. Burrell															A*
300	IB60/NH/1M/NS	1	0															A
300	IB60-300/1M/S	1	30, Red Devil															A

Rh - Reject, high  
Rl - Reject, low  
A - Accept  
F - Fail

\*Borderline but acceptable

TABLE 5. CALIBRATION STANDARDS, HEATED 130°F

PPN	SAMPLE NO.	MONTHS	TIME SHAKEN	MINUTES			Fe	Ag	Al	Cr	Cu	Mg	Na	Ni	Pb	Si	Sn	Ti	Mo	Zn	Status
				1	2	3															
3	B40/15D/6M/S	6	30, Burrell																		A
3	B40/15D/6M/NS	6		0																	A
3	B40/30D/6M/S	6	30, Burrell																		A
3	B40/30D/6M/NS	6		0																	A
3	B40/45D/6M/S	6	30, Burrell																		A
3	B40/45D/6M/NS	6		0																	A
10	B34/15D/8M/S	8	30, Burrell																		A
10	B34/15D/8M/NS	8		0																	A
10	B34/30D/8M/S	8	30, Burrell																		A
10	B34/30D/8M/NS	8		0																	A*
10	B34/45D/8M/S	8	30, Burrell																		R1*
10	B34/45D/8M/NS	8		0																	A*
30	B26/15D/8M/S	8	30, Burrell																		A
30	B26/15D/8M/NS	8		0																	A
30	B26/30D/8M/S	8	30, Burrell																		A
30	B26/30D/8M/NS	8		0																	A
30	B26/45D/8M/S	8	30, Burrell																		A

Rh - Reject, high  
 RI - Reject, low  
 A - Accept  
 F - Fail

\*Borderline but acceptable

TABLE 5. (cont)

P/M	SAMPLE NO.	MONTHS	TIME SHAKEN	MINUTES	TESTS							Zn	Status		
					Fe	Ag	Al	Cr	Cu	Mg	Na	Ni	Pb		
30	B26/45D/8M/NS	8		0											A
50	B25/15D/12M/S	12		-											F
50	B25/15D/12M/NS	12		-	R1										F
50	B25/30D/12M/S	12		-											
50	B25/30D/12M/NS	12		-											
50	B25/45D/12M/S	12		-											
50	B25/45D/12M/NS	12		-											
100	B63/15D/6M/S	6		-											A
100	B63/15D/6M/NS	6		-											A
100	B63/30D/8M/S	8		-	Rh										
100	B63/30D/8M/NS	8		-	Rh										
100	B63/45D/8M/S	8		-											A*
100	B63/45D/8M/NS	8		-											A*
300	IB60/15D/1M/S	1		-											A
300	IB60/15D/1M/NS	1		-											A
300	IB60/30D/1M/S	1		-											A
300	IB60/30D/1M/NS	1		-											A

Rh - Reject, high  
 R1 - Reject, low  
 A - Accept  
 F - Fail

\*Borderline but acceptable

TABLE 5. (cont)

P/N	SAMPLE NO.	MONTHS	TIME SHAKEN												Status		
			MINUTES	Fe	Ag	A1	Cr	Cu	Mg	Na	Ni	Pb	Si	Sn	Ti	Mo	Zn
300	1B60/45D/1M/S	1	-	-	-	-	-	-	-	-	-	-	-	-	-	-	A
300	1B60/45D/1M/NS	1	-	-	-	-	-	-	-	-	-	-	-	-	-	-	A

Rh - Reject, high  
 RI - Reject, low  
 A - Accept  
 F - Fail

\*Borderline but acceptable

F

N

D

D T I C

6 - 8 6